INVESTIGATION OF

BATTERY ACTIVE NICKEL OXIDES

by

P. Ritterman, S. Lerner, and H. Seiger

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOURTH QUARTERLY REPORT

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Solar and Chemical Power Branch
W. J. Nagle

GULTON INDUSTRIES, INC. 212 Durham Ave. Metuchen, N. J.

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INVESTIGATION OF

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SUMMARY

A study of the oxidation state of the sintered nickel oxide electrode, by iodometric titration, shows some agreement with the X-ray data.

A study of the effect of stand, at various temperatures, on structure, was completed.

Positive electrodes with cobalt, and positive electrodes with manganese additives were shown to retain 75% of original capacity after a charged stand at 65° C for 7 days.

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ABSTRACT

Determination of Average Valence of charged plates indicates some agreement with X-ray data.

X-ray patterns of plates after 3 month stands at 10° C, 25° C and 50° C are shown.

Cobalt and manganese doped positives were shown to be superior to pure Ni(OR)₂ controls on charged stand tests.

I. INTRODUCTION

A. PURPOSE

Investigations performed under this contract include the following: (1) identification and characterization of materials formed at the positive electrode of a sintered plate Ni-Cd cell at various temperatures and states of charge: (2) various means of stabilization of the positive electrode material at elevated temperatures; and (3) studies of the utilization efficiency, voltage, and cycling characteristics of the positive electrodes which have been improved by stabilization.

B. STATUS OF PROGRAM AT THE BEGINNING OF THE QUARTER

Further X-ray studies of the sintered plate nickel oxide electrode indicated that no noticeable structure change occurs until the electrode has been at least 90% charged. The structure of the charged state is a disordered Ni(OH)₂ structure. Overcharge causes production of an active material which is completely amorphous to X-ray. Considerable overcharge results in production of NiOOH.

A study of the effect of stand at various temperatures on structure was begun.

Positive electrodes with cobalt additives were shown to retain 75% of original capacity after charged stand at 65°C, for 7 days, as compared to less than 50% for controls.

C. MEETINGS

On March 18, 1966, a meeting was held with Mr. W. Nagle and Mr. H. Schwartz of NASA/Lewis. Dr. R. C. Shair, Dr. H. N. Seiger, Mr. P. Ritterman, and Mr. S. Lerner represented Gulton Industries. Dr. P. Vaughan of Rutgers University, consultant for Gulton Industries, was also present. The meeting took place at Gulton Industries, Metuchen, N. J. The discussion centered around the progress in determining the structure existing in the nickel oxide electrode and the effect of additives on the electrode properties.

II. OUTLINE OF PROGRAM

The program has been divided into two parts. The first involves investigations into the qualitative and quantitative nature of the sintered nickel oxide electrode. These investigations will be carried out by use of chemical, electrochemical, and X-ray diffraction techniques as methods of analysis. The second part concerns itself with methods of stabilizing the charged state of the sintered nickel oxide electrode. The stabilization methods include (1) additives (the inclusion of foreign materials within the structure of nickel oxide crystals), (2) formations at various currents and temperatures, and (3) any other means of stabilization deemed feasible will be presented for approval to the NASA Project Manager. Plates made by these methods will be evaluated with respect to charge retention and performance on charge and discharge.

The details of this program have been discussed in the First, Second and Third Quarterly Reports and are reviewed below.

A. IDENTIFICATION AND CHARACTERIZATION OF BATTERY ACTIVE MATERIAL EXISTING IN THE NICKEL OXIDE ELECTRODES.

1. Variables Investigated

Three effects on active electrode material structure are being studied:

- a. State of charge
- b. Rate of charge
- c. Effect of stand

Stand effects have been investigated at elevated (50°C) , cold (10°C) and room temperatures, both in the presence and absence of the antipode (cadmium electrode).

Three plate cells consisting of single positive plates surrounded by excess negative capacity in the form of two sintered cadmium electrodes are being used for all tests.

2. Methods of Analysis

a. Chemical Means

The formula weight will be determined by a quantitative determination of nickel, hydrogen, and oxygen. A determination of nickel valence will also be made.

(1) Nickel

Total nickel will be determined by electroplating on a platinum grid.

Methods of separating Ni^o from the active nickel oxides have been explored.

(2) Hydrogen

A washed and dried sample will be mixed with $K_2\text{CrO}_4$ and PbCrO₄ and heated to 600°C . The hydrogen in the sample will be converted to water vapor.

(3) Oxygen

Oxygen will be determined by difference with respect to nickel and hydrogen.

(4) Nickel Valence

The valence of nickel will be determined by iodiometric titration and concurrent analysis for active nickel.

b. X-ray Diffraction

All X-ray diffraction data are obtained with uncovered samples. Two goniometer scan speeds are used, one at 2° per minute, the other at $1/8^{\circ}$ per minute.

B. STABILIZATION OF NICKELIC OXIDES AND HYDROXIDES

1. Formation and Various Temperatures and Rates

Formations have been performed at C/2, C/10, and C/30 and temperatures of 10° , 25° , and $80^{\circ}C$ for each rate. These cells were formed at each rate and each temperature for 3 cycles.

Additives

Impregnating nickel solutions have been prepared containing 5%, 10%, and 20% metal atoms of Co, Ba, Mn, Li, Mg, Na, Be, and Ca. These were incorporated by use as the salt of the nitrate ion.

For each quantity and additive type, 10 plates were impregnated.

3. Other Techniques of Stabilization

Other means beside the imprenation used by Gulton Industries will be explored. These will include molten salt impregnation and anodic, as well as cathodic impregnation of sintered plaques from dilute solution of nickel salts.

C. PRELIMINARY EVALUATION

Electrodes stabilized by the above described methods are evaluated in the following manner.

1. Stand

All cells will be charged at the C rate for 2 hours and then immediately discharged at C/5 to 0.0 V.

The charge will be repeated and following a particular stand condition, the cell will be discharged at C/5 to O.O V.

The stand condition will be varied with respect to time and temperature.

Immediate discharge capacity determinations will be made after each stand test.

2. Electrochemical Stabilization

Samples of each group shall be cycled for 4 cycles at C/10 charge--C/5 discharge at room temperature. Cell voltage versus time will be recorded. Cells will be evaluated for capacity, reproducibility of results from cycle to cycle with respect to capacity, efficiency and voltage level.

D. EVALUATION OF IMPROVED MATERIAL

After establishing a superior material, or group of superior materials, the following tests will be made.

1. Decomposition of Charged Positive

Cells prepared with the superior positive will be exposed to stand at temperatures up to 95° C. Charge retention will be obtained as has been described above.

2. Investigation of Sintered Plate Parameters

After establishing a loading factor which meets the approval of the NASA Project Manager, investigation will be made concerning establishment of (a) optimum formation techniques, (b) charge acceptance as a function of charge rate, and determination of point of oxygen evolution at various rates, (c) utilization factors at various discharge rates and utilization factors for depth of discharge from 25% to 100%.

Details of these tests have been described in the previous Quarterly Reports.

III. EXPERIMENTAL PROCEDURES & RESULTS

A. EXPERIMENTAL PROCEDURES

1. Determination of Battery Active Material Existing in the Sintered Plate Nickel Oxide Electrode

a. Effect of State of Charge

In the previous quarter, it was determined that the structure of the positive plate was unchanged between the fully discharged and the 90% charged state, and that the major structural changes occur at full charge and at overcharge.

During this quarter, positive plates in various states of charge and overcharge were studied by means of iodometric titration (1) in order to determine the valence state of the active material. The determinations were performed in the following manner: Cells consisting of one positive (whose active material content was known) and two negative plates were assembled and charged to the required state of charge. The cells were then disassembled, the positives removed, rinsed in distilled water, cut into small pieces, and the titration performed as described below.

Positive electrodes, in the following states of charge, were used: 50%, 100% (2 determinations) 300% and 500%.

b. Effect of Stand Time at Various Temperatures

(1) Construction of Cells

Thirty cells, consisting of 1 positive and 2 negatives, were assembled. Each positive plate had dimensions of 1-7/8" x 2-1/8" x .035" and contained 3.4 grams of Ni(OH)₂.

(2) Formation Procedure

All cells were formed at 100 ma. The charge lasted for 15 hours, then discharged until the cells reached zero volts. Upon completion of discharge, the cells were shorted for 2 hours. This procedure was repeated twice more.

(3) Selection of Cells and Test Procedures

Based on the capacity of the third formation cycle (Table II), 24 cells, most alike in capacity, were charged at 100 ma for 20 hours at room temperature. Twelve of the cells were then disassembled in the charged state and their positives placed in beakers of 34% potassium hydroxide. The other 12 cells remained as they were. Four plates and four cells were placed in an oven at 50°C.

A like number of cells and plates were placed in a cold box at 10°C, and the remaining cells and plates were kept at room temperature. After a stand of 24 hours, a plate and a cell were removed from each temperature ambient, the cells disassembled, and all plates X-rayed. After 48 hours, another cell and corresponding plates were removed from each ambient and the positive plates X-rayed. The remaining cells and plates were retained at their respective temperatures for a three month period, at which time the positives were X-rayed.

c. J-NiOOH (Ni₂O₃·H₂O)

With the hope of preparing an X-ray standard, a compound was prepared by the method of Cairns and Ott (2) which was analyzed by them and found to have the composition of Ni₂O₃ • H₂O_•. This compound is a dimer of NiOOH and the reported X-ray data are similar to that given by Glemser and Einerhand (3) for 6-NiOOH.

d. Oxidation State of Nickel

The oxidation state of nickel in the compound described above was determined in the following manner (1).

The sample was placed in a beaker of alkaline KI. The sample was then dissolved by addition of dilute sulfuric acid until the pH reached 1.5. The iodine liberated by the reduction of nickel in oxidation states greater than 2 to Ni $^{++}$ is titrated with a standard solution of Na₂S₂O₃. A sintered plate was also analyzed by this method.

2. Stabilization

a. Additives

(1) Preparation of plates and cells

Solutions were prepared in which the concentration of nickel ions were decreased and replaced by other cations in such a manner that the other cations were present to the extent of 20 atom percent. Ten plates were prepared for each of the solutions. The other cations were: magnesium, manganese, lithium, sodium. Ten control plates were also prepared. Each plate was impregnated to a weight gain of approximately 3 grams. The plates were then built into six groups of ten cells each. Each group was further subdivided into two subgroups; one subgroup of four cells to be used for cycling experiments, and the other subgroup containing six cells to be used for charged-stand tests.

(2) Effect of Cycling

Four cells from each group were cycled 4 times. Each charge was for a period of 16 hours at 100 mA, followed immediately by a discharge at 200 ma to a cell voltage of 0.0 V. All cells were shorted overnight between cycles.

(3) Effect of Stand

The remaining six cells from each group were charged at 1 ampere for two hours and were then immediately discharged to 0.0 V at 200 mA at room temperature. The cells were then shorted overnight and again charged at 1 ampere for 2 hours. The cells were allowed to stand in the charged state for 3 days at room temperature; following this stand, the cells were discharged at 200 mA to 0.0 V and shorted overnight. This cycle was repeated for a 3 day stand at 65°C and again for a 7 day stand at 65°C for a group of cells containing selected additives.

B. EXPERIMENTAL DATA

1. Determination of Battery Active Materials Existing in the Sintered Plate Nickel Oxide Electrode

a. Effect of State of Charge

Table I lists the results of the valence determinations on sintered nickel oxide electrodes at various states of charge along with that of the chemically prepared -NiOOH (Ni₂O₃ • $\rm H_2O$).

b. Effect of Stand at Various Temperatures

The capacities of the 30 cells on their final formation discharge are listed in Table II. Numbers marked with an asterisk indicate the 24 cells chosen.

Figures 1, 2, 5, 6, 7, and 8 are X-ray patterns obtained from stored plate positives. Figures 3, 4, 9 and 10 are X-ray patterns obtained from stored cell positives. Figures 1, 2, 3 and 4 are from plates stored at 10°C. Figures 5 and 6 are from plates stored at room temperature and Figures 7, 8, 9 and 10 are from plates stored at 50°C. All plates were stored at their respective temperature for 3 months.

c. 7-NiOOH (Ni₂O₃ - H₂O)

The X-ray pattern for this compound is shown in condensed form in Figure 11 and the X-ray "d" spacings and integrated intensities are shown in Table III. The integrated intensities were obtained by measuring the areas under the peaks with a mechanical integrator.

The oxidation state of the nickel oxide was determined to be $2.79 \pm 20\%$. The data for the determination is shown in Table I.

2. Stabilization of Nickelic Oxides and Hydroxides

a. Additives

(1) Preparation of Plates

Tables IV through X list the weight gain, theoretical capacity and experimental capacity on the third formation cycle of cells with positives containing, respectively, 20 metal atom percent of beryllium, magnesium, lithium, sodium, manganese, and cobalt, along with the control cells.

(2) Effect of Cycling

Table XI shows the capacities of 4 cells in each group for 4 consecutive cycles. Figure 12 shows a typical discharge curve for each group.

(3) Effect of Stand

Table XII shows the capacities of 6 positives from each additive and control group on immediate discharge, 3 day stand at 25°C, 3 day stand at 65°C, and for selected additives on 7 day stand at 65°C. The data for the positives containing cobalt are for 1 and 7 days at 65°C, taken from the Third Quarterly Report and included here to complete the table.

C. DISCUSSION

1. Determination of Battery Active Materials Existing in the Sintered Plate Nickel Oxide Electrode

a. Effect of State of Charge

(1) Oxidation State of Active Material

From the values of the oxidation state, shown in Table I, and the duplicate determination of one of the charged states, it is apparent that the fraction of trivalent nickel is precise to + 20%. At the 50% charge level, the composition of the electrode is 34% NiOOH - 66% Ni(OH)₂, and at the fully charged (100%) level, the composition is 45% NiOOH - 55% Ni(OH)₂. The composition at 500% charged, at which state the electrode has previously been shown to be composed of J-NiOOH(4), is 75% J-NiOOH - 25% Ni(OH)₂ or an average valence of 2.75. This is in favorable agreement with the value of 2.79 obtained for the J-NiOOH (Ni₂O₃ · H₂O) prepared in the laboratory.

However, the valence determinations are not in agreement with the electrochemical data which indicate that, at high charge rates, a cell charged to 100% of capacity will, on discharge, give back between 85% and 90% of its rated capacity. This discrepancy between the electrochemical and chemical data is, as yet, unexplained. During the Fifth Quarter, work will be initiated in order to try and resolve this discrepancy.

b. Effect of Stand on Structure

The results of Figures 1 through 10 are summarized in Table XIII with respect to an estimate of the intensities of the reflections of the 001, 100 and 101 planes of the Ni(OH)₂ structure, corresponding to the nominal "d" values 4.62, 2.73 and 2.32 Å. The intensities are given as a function of area under the curve. The results show that at 10° and 50°C, except for Figure 4 which appears to be anomalous, that there is considerably less self discharge in positive electrodes stored without their antipodes (negative electrodes) than those positives stored as cells. Since it was not possible to locate the two positive electrodes stored as cells at room temperature, it is not possible to obtain any quantitative data on the self discharge as a cell versus that of a single electrode. However, it is assumed that the same results should apply at 25°C as were found at 10° and 50°C.

2. Stabilization of Nickelic Oxides and Hydroxides

a. Additives

Figure 13, which is a graphical summary of the 65°C stand shown in Table XII, clearly shows the superiority of charge retention by both the cobalt and manganese additions to the positives over all other additions and the control positives.

IV. EVALUATION OF IMPROVED MATERIALS

With their superiority, with respect to high temperature charge retention, established, positive electrodes containing 20 metal atom percent cobalt and 20 metal atom percent manganese were selected as improved materials and, along with control cells, were subjected to further evaluation.

A. UTILIZATION FACTORS OF VARIOUS RATES OF DISCHARGE

1. Experimental Procedure

Four cells with cobalt containing positives, four with manganese containing positives, and four controls were chosen. The cells were charged at 1 ampere for two hours and then discharged at the 6C rate to 0.0 V and shorted overnight. This procedure was repeated except that the discharge rate was changed to the C rate, C/2 rate, C/5 rate and C/10 rate.

2. Results

The results of the above experiment are summarized in Table XIV and shown graphically in Figures 14 through 17.

3. Discussion

For all rates of discharge, the capacity of the cobalt containing positives is higher than that of the manganese and control positives. The control positives have, again, a somewhat higher capacity than the manganese containing positives. In Figure 18 there is a plot of percent change in capacity using the value at 100 ma for normalization versus discharge rate. The effect of charge rate on these electrodes is similar. In fact, the manganese and control electrodes are essentially identical, while the slope for the cobalt containing plates is less than that of the other two. Between the amount of capacity and the slope for the discharge rate, plates with cobalt are, indeed, attractive.

V. CONCLUSIONS

- 1. The use of the iodometric titration method to determine the oxidation state of the charged nickel oxide electrode has shown itself to be applicable. However, further refinement of the technique is indicated.
- 2. Charged positive electrodes show a greater charge retention (as determined by X-ray methods), at all stand temperatures, when allowed to stand without their antipodes, than as assembled cells.
- 3. The charge retention and utilization characteristics of cobalt and manganese additions in positives are superior to those of pure Ni(OH)₂ positives and positives containing other studied additives.
- 4. Cobalt containing positives have significantly higher capacities, for the same weight gain, than pure Ni(OH)₂ positives.

VI. FUTURE WORK

- A. A small cell will be constructed which will allow the positive to be X-rayed while under continuous charge.
- B. The technique to enable the method of determining the oxidation state of nickel to be applied to the sintered positive electrode will be further investigated.
- C. A technique to study the sintered electrode by infrared analysis will be investigated.
- D. Sintered nickel positives containing additives at other than the 20 atom percent level will be evaluated.

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- (3) O. Glemser and J. Einerhand, Zeit. fur Anorg. Chem. 261, 43, (1950).
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TABLE I

DETERMINATION OF AVERAGE VALENCE OF SINTERED NICKEL OXIDE ELECTRODES AT VARIOUS STATES OF CHARGE

PERCENT CHARGED	WEIGHT-ACTIVE NICKEL (gms)	MILLIEQUIVALENTS ACTIVE NICKEL	MILLIEQUIVALENTS Na ₂ S ₂ O ₃ = MILLIEQUIV. Ni ³⁺	MILLIEQUIVALENTS N1 MILLIEQUIVALENTS N1	AVERAGE
50	1.22	20.85	7.06	76.0	2.34
100	1.19	20.32	9.93	0.49	2.49
100	1.21	20.54	8,34	0.41	2.41
300	1.16	19.70	11.60	09.0	2.60
200	1,29	21.84	16.34	0.75	2.75
(N1 ₂ 0 ₃ ·H ₂ 0)	0.13	2.18	1.72	0.79	2.79

TABLE II

CAPACITY ON FINAL FORMATION - STAND CELLS

CELL	CAPACITY		CELL	CAPACITY		
NUMBER	To 1.0 V	To 0.0 V	NUMBER	To 1.0V	To 0.0 y	
	.98	.98	• 16	.90	.91	
2	1.03	1.03	* 17	.93	.93	
3	.93	.94	* 18	.94	.95	
4 militaria. Tanàna Tanàna mandriana	.97	.97	* 19	.94	.95	
5	9 5	**************************************	20	.65	.12	
6		.98	* 21	.92	.	
7	1.05	1.01	22	1.03	1.4	
8	1.04		3 23	.96		
9	.95	.95	* 24	1.00	1.00	
10	.99	.99	* 25	.97	.97	
11	.98	.98	* 26	.96	.96	
12	.91	.92	* 27	.98	.98	
13	1.00	1.00	* 28	1.01	1.01	
14	.85	.85	* 29	.97	.97	
15	.83	.8 3	* 30	.96	.9 6	

^{*} Cells Chosen - Most alike in capacity

TABLE III

X-RAY DIFFRACTION PATTERN X-NiOOH (Ni₂O₃·H₂O)

1					
	2 0	d	I/I _o	hkl (%-NiOOH)	MATERIAL
	12.3	7.19	100в	003	У-NiOOH - Ni ₂ O ₃ • H ₂ O
	24.9	3.57	18в	006	У-nioon - ni ₂ o ₃ • н ₂ o
	33.3	2.69	14B		Ni ₂ 0 ₃ • H ₂ 0
	36.5	2.47	8в	101	У-N100H - N1 ₂ 0 ₃ ⋅ H ₂ 0
	38.0	2.37	12B	102	δ-ni00H - ni ₂ 0 ₃ • H ₂ 0

B = Broad

FORMATION CAPACITIES OF CELLS WITH POSITIVES CONTAINING
20 METAL ATOM % BERYLLIUM

f			DISCHARGED AT 100 ma			
CELL	TOTAL WT.	WEIGHT		MENTAL	THEORETICAL CAPACITY BASED	
NO.	GAIN	Ni(OH) ₂		CITY	ON WEIGHT N1(OH) ₂	
	(gms)	(gms)	1.0 V	0.0 V	0.0 V	
1	2.98	2.72	0.75 AH	0.76 AH	0.79 AH	
2	3.11	2.84	0.75	0.75	0.82	
3	2.99	2.73	0.74	0.75	0.79	
4	3.09	2.82	0.75	0.76	0.82	
5	3.04	2.78	0.75	0.78	0.80	
6	3.06	2.80	0.76	0.78	0.81	
7.	3.00	2.74	0.75	0.77	0.79	
8	2.94	2.69	0.75	0.76	0.78	
9	3.07	2.81	0.75	0.80	0.81	
10	2.95	2.70	0.75	0.78	0.78	

FORMATION CAPACITIES OF POSITIVES CONTAINING
20 METAL ATOM % MAGNESIUM

T			DISCHARGED AT 100 ma				
CELL	TOTAL WT.	WEIGHT		RIMENTAL	THEORETICAL CAPACITY BASED		
NO.	GAIN	Ni(OH) ₂		PACITY	ON WEIGHT Ni(OH) ₂		
	(gms)	(gms)	1.0 V	0.0 V	0.0 V		
1	3.05	2.67	0.67 AH	0.68 AH	0.77 AH		
2	3.06	2.67	0.66	0.67	0.77		
3	3.00	2.62	0.65	0.66	0.76		
4	3.06	2.67	0.68	0.70	0.77		
5	3.06	2.67	0.68	0.71	0.77		
6	3.02	2.64	0.67	0.68	0.76		
7	3.10	2.71	0.67	0.69	0.79		
. 8	3.01	2.63	0.66	0.69	0.76		
9	3.01	2.63	0.66	0.67	0.76		
10	2.99	2.63	0.68	0.70	0.76		
		.]				

FORMATION CAPACITIES OF CELLS WITH POSITIVES CONTAINING
20 METAL ATOM PERCENT LITHIUM

CETT	TOTAL THE			DISCHARGED AT 100 ma			
CELL TOTAL WI.		-,,		RIMENTAL	THEORETICAL CAPACITY BASED		
110.	(gms)	Ni(OH) ₂ (gms)		PACITY	ON WEIGHT N1(OH)2		
	(Bille)	(Ems)	1.0 V	0.00	0.0V		
1	3.07	2.91	0.95 AH	0.95 AH	0.84 AH		
2	2.96	2.81	0.91	0.91	0.81		
3	3.04	2.88	0.93	0.94	0.83		
4	3.05	2.89	0.90	0.91	0.84		
5	3.06	2.90	0.93	0.94	0.84		
6	2.97	2.82	0.92	0.93	0.82		
7	2.97	2.82	0.90	0.91	0.82		
8	2.99	2.83	0.91	0.92	0.82		
9	2.97	2,82	0.93	0.93	0.82		
10	2.98	2.83	0.90	0.92	0.82		

FORMATION CAPACITIES OF CELLS WITH POSITIVES CONTAINING 20 METAL ATOM PERCENT SODIUM

TABLE VII

	TOTAL WT.	WEIGHT Ni(OH) ₂	DISCHARGED AT 100 ma					
CELL NO.			CAPA	MENTAL CITY	THEORETICAL CAPACITY BASED ON WEIGHT Ni(OH)2			
	(gms)	(gms)	1.0 V	0.0	0.0 V			
1	3.03	2.77	0.94 AH	0.96 AH	0.80 AH			
2	3.10	2.83	0.91	0.92	0.82			
3	2.99	2.73	0.89	0.92	0.79			
4	3.12	2.85	0.92	0.93	0.82			
5	3.03	2.77	0.87	0.87	0.80			
6	2.99	2.73	0.94	0.95	0.79			
7	3.00	2.74	0.89	0.92	0.79			
8 .	2.96	2.71	0.95	0.96	0.78			
9	3.05	2.79	0.93	0.94	0.80			
10	2.95	2.70	0.89	0.91	0.78			

TABLE VIII.

FORMATION CAPACITIES OF CELLS WITH POSITIVES CONTAINING 20 METAL PERCENT MANGANESE

				DISCHARG	ED AT 100 ma
CELL	TOTAL WT.	WEIGHT		MENTAL	THEORETICAL CAPACITY BASED
NO.	GAIN	Ni(OH) ₂		CITY	ON WEIGHT Ni(OH) ₂
	(gms)	(gms)	1,0 V	0.0 V	0.0 V
1	3.20	2.58	0.82 AH	0.88 AH	0.75 AH
2	3.09	2.50	0.81	0.87	0.72
3	3.13	2.53	0.82	0.88	0.73
4	3.07	2.48	0.81	0.87	0.72
5	3.04	2.47	0.78	0.85	0.71
6	3.27	2,64	0.85	0.88	0.76
7	3.08	2.49	0.81	0.86	0.72
8 ·	3.17	2.57	0.84	0.87	0.74
9	3.11	2.51	0.81	0.86	0.73
10	3.07	2.48	0.80	0.85	0.72
	÷				

FORMATION CAPACITIES OF CELLS WITH POSITIVES CONTAINING
20 METAL ATOM PERCENT COBALT

TABLE IX

CELL NO.	TOTAL WT. GAIN (gms)	WEIGHT Ni(OH) ₂ (gms)	1	MENTAL ACITY	THEORETICAL CAPACITY BASED ON WEIGHT N1(OH)2
	\Q\	Ema	1.0 V	0.0 V	0.0 V
1	3,13	2.50	0.91 AH	0.92 AH	0.72 AH
2	3.09	2.47	0.90	0.90	0.71
. 3	2.95	2.36	0.87	0.87	0.68
4	2.90	2.32	0.85	0.85	0.67
5	2,96	2.37	0.86	0.86	0.68
6	3.17	2.54	0.92	0.93	0.73
7	3.15	2.52	0.92	0.93	0.73
8	2.88	2.30	0.83	0.84	0.66
9	2.95	2.36	0.87	0.87	0.68
10	3.13	2.50	0.92	0.93	0.72

TABLE X

FORMATION CAPACITIES - CONTROL CELLS

		DISCHARGED AT 100 ma							
NO.	WEIGHT Ni(OH) ₂		NTAL CAPACITY	THEORETICAL CAPACITY BASED ON WEIGHT N1 (OH) 2					
	(gms) ²	1.0 V	0.0 V	0.0 V					
1	2.98	0.92 AH	0.93 AH	0.86 AH					
2	3.00	0.92	0.93	0.87					
3	3.15	0.93	0.94	0.91					
4	3.06	0.90	0.91	0.88					
5	3.04	0.92	0.93	0.88					
6	3.03	0.93	0.93	0.88					
7	3.12	0.93	0.93	0.90					
8	3.01	0.94	0.94	0.87					
9	3.00	0.93	0.94	0.87					
10	2.97	0.89	0.90	0.86					

CYCLING DATA FOR CELLS WITH POSITIVES CONTAINING 20 ATOM % METAL AND CONTROL CELLS

				HARGED AT				
CELL	CYCLE I		CYCLE II		CYCLE III		CYCLE	IV
NUMBER	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V	0.0 V
n. 1	0 -0							
Be-1	0.79 AH	0.85 AH		0.85 AH	0.81 AH	0.86 AH	0.81 AH	0.87 AH
Be-2	0.81	0.84	0.84	0.88	0,82	0.85	0.82	0.85
Be-3	0.78	0.83	0.79	0.83	0.78	0.82	0.78	0.82
Be-4	0.81	0.85	0.82	0.84	0.82	0.85	0.82	0.86
Mg-1	0.72	0.74	0.68	0.71	0.64	0.67	0.59	0.62
Mg-2	0.68	0.72	0.65	0.69	0.61	0.68	1 - 1	
Mg-3	0.70	0.72	0.65	C.68	0.61	0.65	0.58	0.63
Mg-4	0.71	0.72	1	1	13		0.58	0.59
mg-4	0.71	0.72	Short	Short	0.67	0.69	0.59	0.62
Li-1	0.94	0.96	0.97	0.98	0.96	1.00	0.99	1.03
Li-2	0.93	0.94	0.97	0.97	0.94	0.99	0.97	1.01
Li-3	0.93	0.95	0.96	0.97	0.94	0.94	0.97	1.01
Li-4	0.90	0.92	0.93	0.97	0.92	0.97	0.96	1.00
Na-1	0.94	0.97	0.93	1.06	0.91	1.04	0.06	1 02
Na-1 Na-2	0.88	0.95			ti -	1.04	0.96	1.02
			0.86	0.95	0.86	0.97	0.88	0.99
Na-3	0.89	0.96	0.86	0.96	0.86	0.98	0.89	1.00
Na-4	0.93	0.97	0.88	1.01	0.84	0.96	0.87	0.98
Mn-1	0.89	0.98	0.97	0.98	0.89	0.97	0.87	0.94
Mn-2	0.90	0.97	0.90	0.96	0.87	0.95	0.85	0.93
Mn-3	0.90	0.99	0.90	0.97	0.87	0.96	0.85	0.93
Mn-4	0.88	0.96	0.88	0.95	0.87	0.94	0.84	0.92
Co-6	0.85	0.88	0.87	0.89	0.91	0.94	0.91	0.94
Co-7	0.89	0.91	0.92	0.95	0.91	0.94	0.96	0.94
.					11		0.96	0.98
Co-8	0.88	0.91	0.91	0.93	0.95	0.99		
Co-9	0.82	0.84	0.85	0.87	0.91	0.94	0.91	0.92
Control-11	0.94	1.07	0.92	1.06	0.92	1.06	0.94	1.04
Control-12	0.90	1.01	0.90	1.00	0.88	1.03	0.92	1.02
Control-13	0.92	1.01	0.92	1.01	0.91	1.03	0.94	1.02
Control-14	0.90	0.97	0.86	0.95	0.84	0.92	0.86	0.93
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TABLE XII

CAPACITIES OF CELLS WITH POSITIVES CONTAINING 20 ATOM % METAL ADDITIVES AND CONTROL CELLS AT VARIOUS TEMPERATURES

	DISCHARGED AT 200 ma							
CELL	NO ST	AND	3 DAYS		3 DAYS	65°C	7 DAYS	65°C
NUMBER	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V		1.0 V	0.0 V
Be-5	0.83 AH	0.87 AH	0.77 AH	0.80 AH		0.56 AH		
Be-6	0.79	0.84	0.72	0.75	0.55	0.56		
Be-7	0.75	0.84	0.73	0.74	0.56	0.57		
Be-8	0.77	0.80	0.72	0.73	0.59	0.59		
Be-9	0.79	0.84	0.74	0.77	0.58	0.58		
Be-10	0.77	0.86	0.77	0.78	0.59	0.59		
	1		1					
			_					
Mg-5	0.89	0.93	0.73	0.75	0.65	0.65		
Mg-6	0.88	0.93	0.72	0.73	0.57	0.57		
Mg-7	0.86	0.88	0.69	0.69	0.53	0.53		
Mg-8	0.89	0.93	0.72	0.73	0.56	0.56		
Mg-9	0.90	0.95	0.73	0.75	0.61	0.63		
Mg-10	0.90	0.95	0.77	0.78	0.60	0.62		
Li-5	1.13	1.19	1.03	1.03	0.75	0.83	0.27 AH	0.28 AH
Li-6	1.14	1.21	1.06	1.07	0.66	0.85	0.28	0.29
Li-7	1.04	1.07	0.94	0.94	0.69	0.86	0.27	0.28
Li-8	1.13	1.17	1.05	1.05	0.66	0.89	0.27	0.28
Li-9	1.19	1.24	1.00	1.00	0.90	0.95	0 22	0 24
Li-10	1.12	1.17	1.05	1.06	0.81	0.86	0.33	0.34
Na-5	0.97	0.99	0.81	0.82	0.48	0.52		
Na-6	0.96	0.99	1.00	1.00	0.74	0.77		
Na -7	1.01	1.03	0.94	0.94	0.66	0.73		
Na-8	0.85	0.96	1.01	1.01	0.85	0.90		
Na-9	1.00	1.04	0.83	0.83	0.03	0.80		
Na-10	0.93	0.97	0.87	0.88	0.74	0.77	,	
Na-10	0.55	0.57	0.07	0.00	0.,4	0.,,		
				}	1			
Mn-5	0.88	0.98	0.82	0.93	0.95	0.95	0.54	0.72
Mn-6	0.97	1.05	0.93	0.98	0.86	0.87	0.71	0.80
Mn-7	0.93	1.03	0.88	0.96	0.82	0.85	0.70	0.78
Mn-8	0.95	1.03	0.89	0.95	0.85	0.85	0.76	0.76
Mn-9	0.94	1.03	0.89	0.97	0.85	0.85	0.79	0.82
Mn-10	0.94	1.02	0.88	0.92	0.73	0.75	0.47	0.48
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....Continued

TABLE XII - CONTINUED

CELL		STAND	3 DAYS	25°C	3 DAYS	65°C	7 DAYS	65°C
NUMBER	1.0 V	0.0 Y	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V	0.0 V
Co-1	1.15AH	1.25AH			1.02 (1 day)	1.03 (1 day)	0.88AH	0.92AH
Co-2	1.16	1.28			1.04 "	1.08 "	0.92	0.94
Co-3	0.98	1.07			0.93 "	0.94 "	0.72	0.77
Co-4	1.00	1.07			0.95 "	0.97 "	0.72	0.81
Control-15	1.09	1.09	1.03	1.03	0.75	0.80	0.52	0.57
Control-16	1.11	1.11	1.06	1.06	0.78	0.85	0.63	0.70
Control-17	1.06	1.06	1.00	1.01	0.72	0.81	0.58	0.70
Control-18	1.08	1.08	1.06	1.06	0.93	0.97	0.80	0.85
Control-19	1.11	1.11	1.06	1.07	0.90	0.94	0.45	0.45
Control-20	1.05	1.06	1.02	1.03	0.88	0.92	0.80	0.89
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						.	1	

TABLE XII - CONTINUED

	DISCHARGED AT 200 ma									
CELL	NO	STAND	3 DAYS	25°C	3 DAYS	65°C	7 DAYS	65°C		
NUMBER	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V	0.0 V	1.0 V	0.0 V		
Co-1	1.15AH	1.25AH			1.02 (1 day)	1.03 (1 day)	HA88.0	0.92AH		
Co-2	1.16	1.28			1.04 "	1.08 "	0.92	0.94		
Co-3	0.98	1.07			0.93 "	0.94 "	0.72	0.77		
Co-4	1.00	1.07			0.95 "	0.97 "	0.72	0.81		
)}			
-							1			
Control-15	1.09	1.09	1.03	1.03	0.75	0.80	0.52	0.57		
Control-16	1.11	1.11	1.06	1.06	0.78	0.85	0.63	0.70		
Control-17	1.06	1.06	1.00	1.01	0.72	0.81	0.58	0.70		
Control-18	1.08	1.08	1.06	1.06	0.93	0.97	0.80	0.85		
Control-19	1.11	1.11	1.06	1.07	0.90	0.94	0.45	0.45		
Control-20	1.05	1.06	1.02	1.03	0.88	0.92	0.80	0.89		
				1			1			
							#			
				-						
	}									
				l						

TABLE XIII

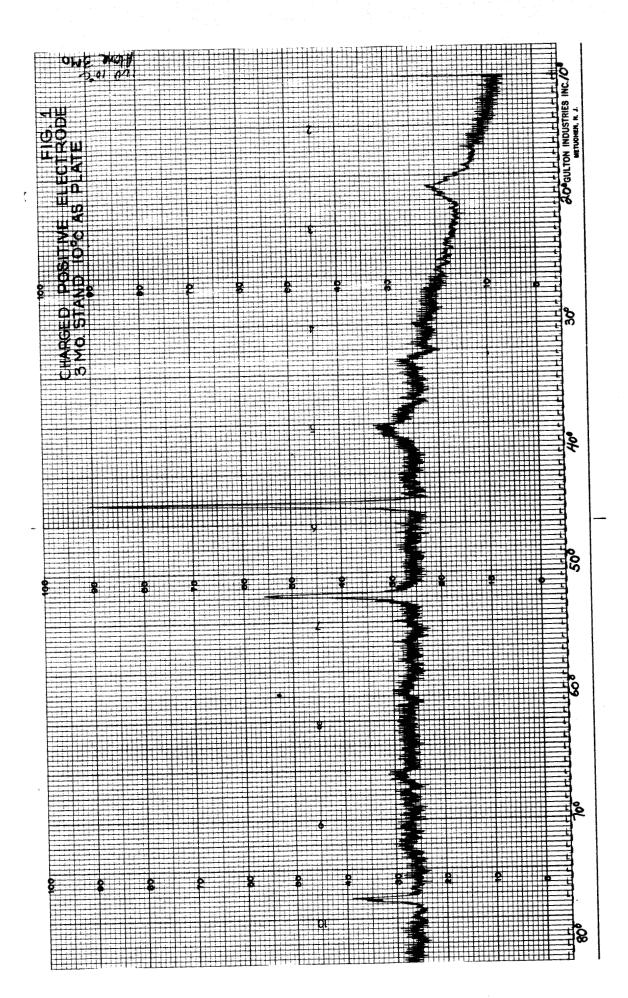
X-RAY DIFFRACTION LINES AND THEIR INTEGRATED INTENSITIES OBTAINED FROM POSITIVE PLATES STORED FOR THREE MONTHS AT VARIOUS TEMPERATURES

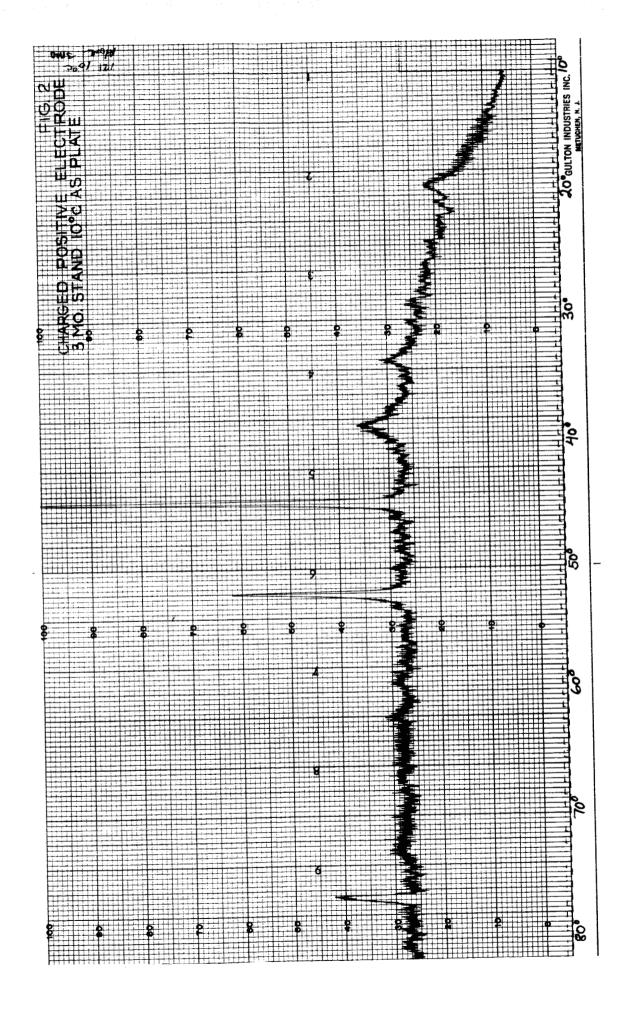
FIGURE	STORED		X-RAY "d" SPACINGS & INTENSITIES								
NO.	AS	TEMPERATURE									
1	Plate	10 ⁰ C	4.77 (8)	2.71 (4)	2,34 (12)						
2	Plate	10 ⁰ C	4.62 (8)	2.68 (4)	2.36 (12)						
3	Cell	10 ⁰ C	4.62 (10)	2.70 (15)	2.34 (17)						
4	Cell	10°C	4.60 (4)	2.71 (2)	2.33 (12)						
5	Plate	25°C	4.55 (5)	. 2.68 (3)	2.32 (10)						
6	Plate	25 ⁰ C	4.74 (4)	2.73 (4)	2.34 (15)						
7.	Plate	50°C	4.60 (3)	2.69 (5)	2.34 (12)						
8	Plate	50°C	4.55 (3)	2.71 (2)	2,32 (5)						
9	Cell	50°C	4.57 (10)	2.71 (8)	2.33 (18)						
10	Cell	50°C	4.60 (15)	2.71 (10)	2.32 (18)						
			<u> </u>								

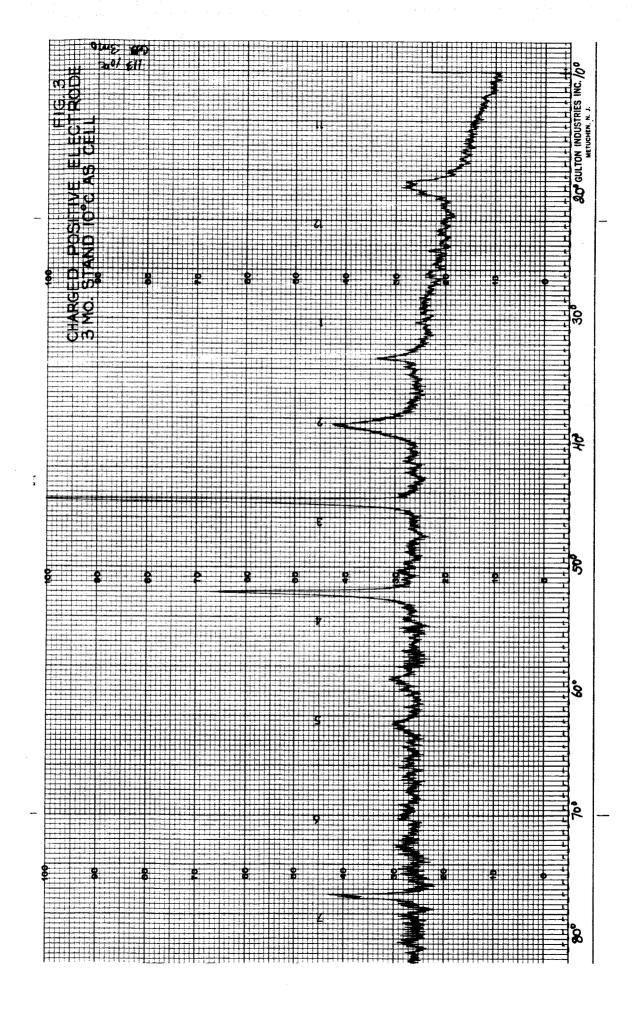
TABLE XIV

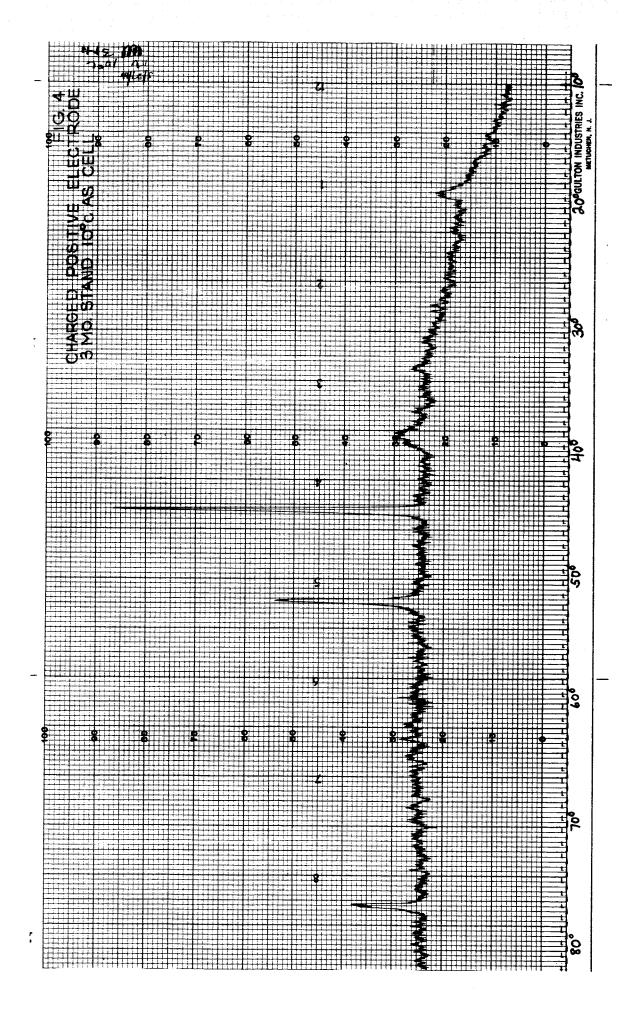
UTILIZATION FACTORS AT VARIOUS RATES OF DISCHARGE

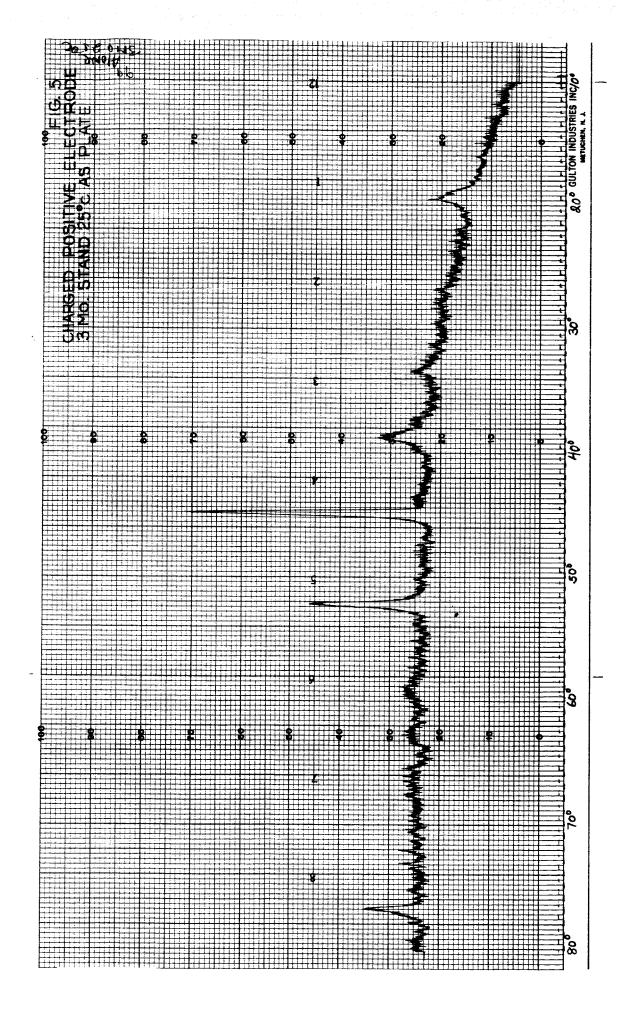
_	_						 				 				
	(0.1 A)	Λ 0.0	1.17	1.12	1.07	1.08	1,32	1.25	1,26	1,23	1,15	1.16	1,13	1.06	
	c/10 (Λ 0•1	66	.97	68	* 6*				1.17		1.03		.95	
	(0.2 A)	0.0 V	76.	95	96	96			1,23	1,22	1.09	1.09	1.07	1.01	
	1	1.0 V	.75	.78	.75	.75			1,15		.85	88.	.87	.83	
	.5 A)	Λ 0.0	.79	.83	•79	.83	1.12	1.06	1.06	1.02	.85	.85	.82	.77	
RATE	c/2 (0	I .0 V	.67	.71	.63	89•	101	.83	.93	88	 .73	.71	.75	99•	
	(1.0 A)	Λ 0 0	68	.95	-89	.95	1,22	1.14	1.17	1,15	.92	.92	.92	85	
) ວ	1.0 V	.67	.75	.67	.67	1.00	.85	.92	.85	 .80	80	.80	.67	
	6C (6.0 A)	0°0 V	.50	09.	• 50	.50	96.	.60	1.00	1,00	09.	09.	09•	09•	
) 29	1.0 V	:	!	!	1	1	1	1	: :	:	!!	1 1	!	
	CELL	NUMBER	Mn-1	Mn-2	Mn-3	Mn-4	Co-11	Co-12	Co-13	Co-14	 Control-11	Control-12	Control-13	Control -14	

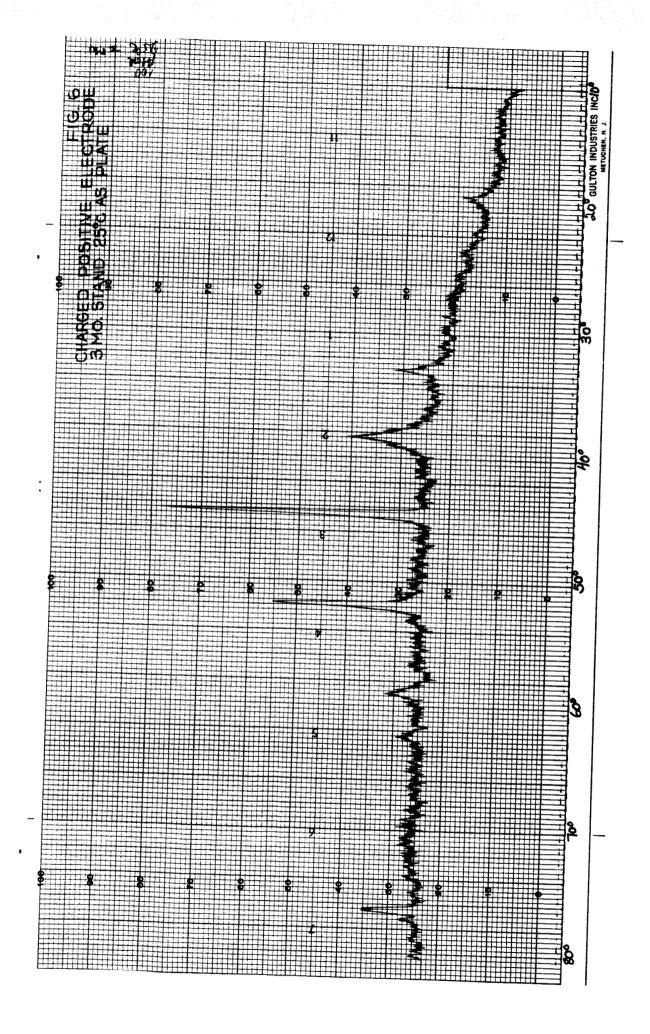


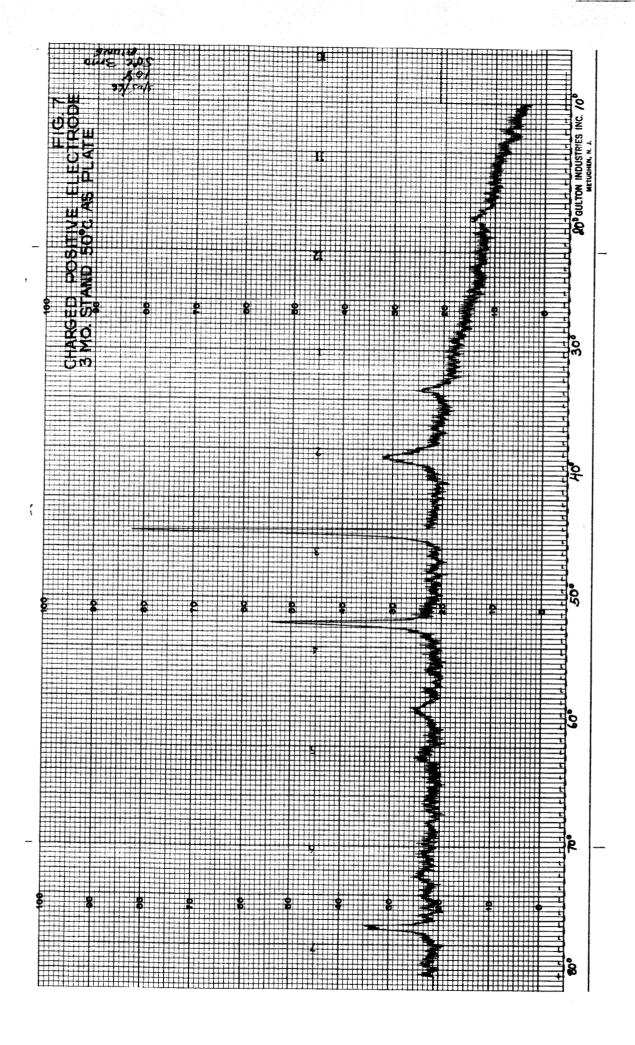


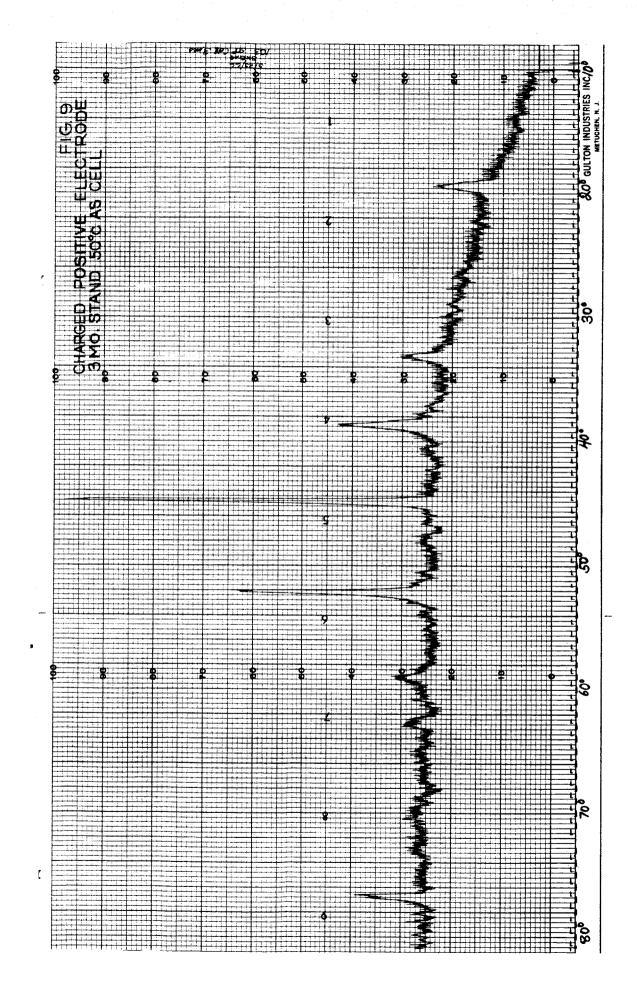


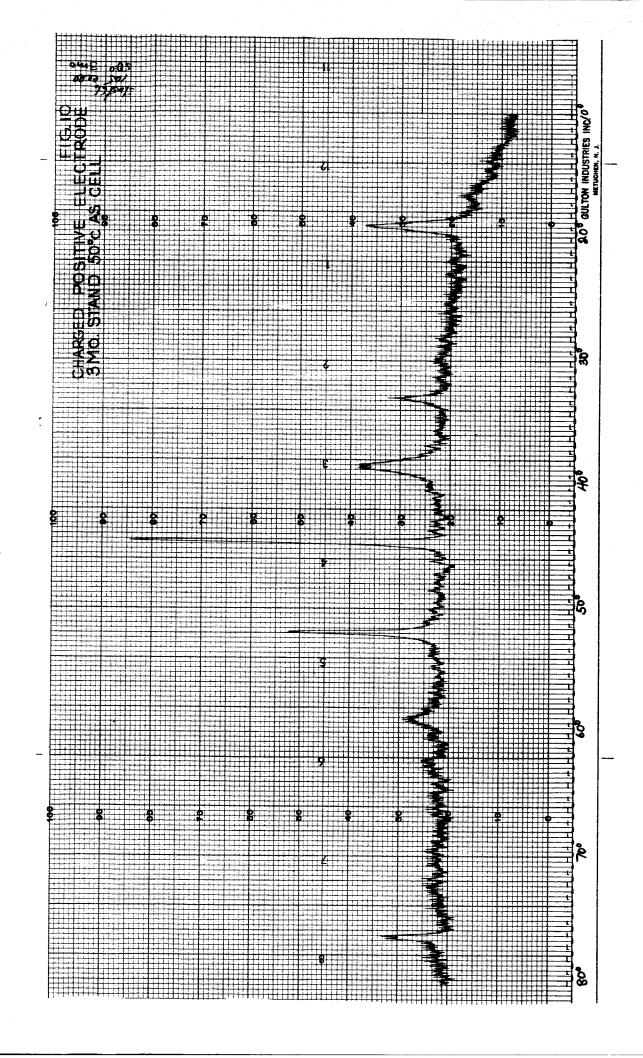


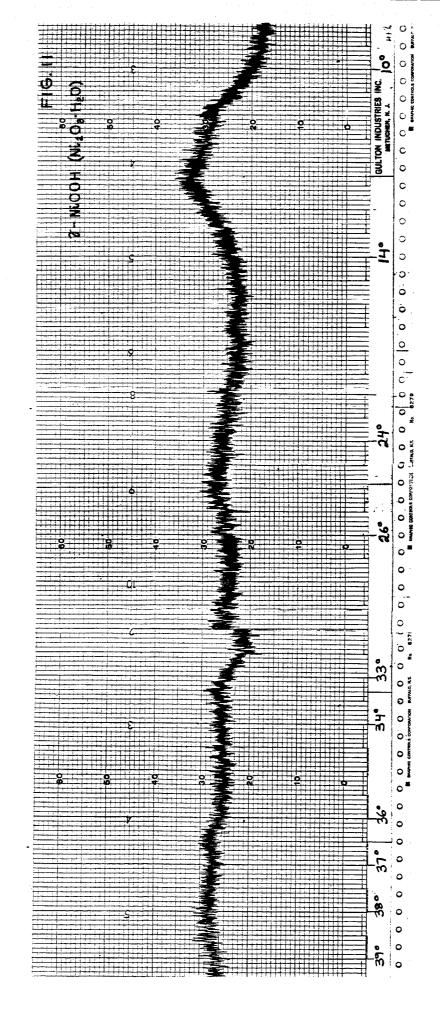


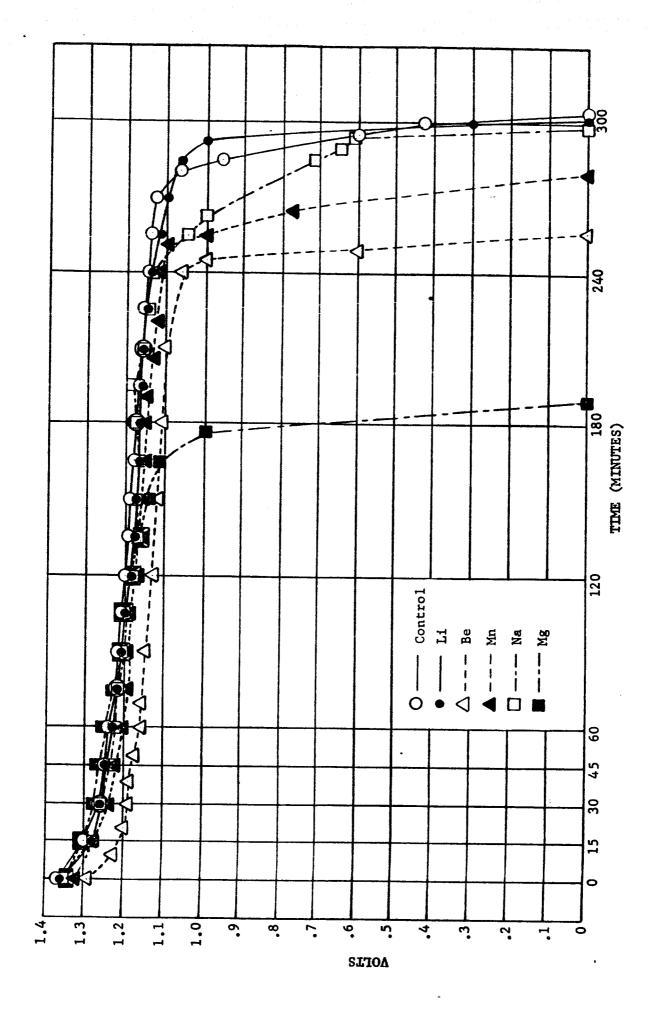




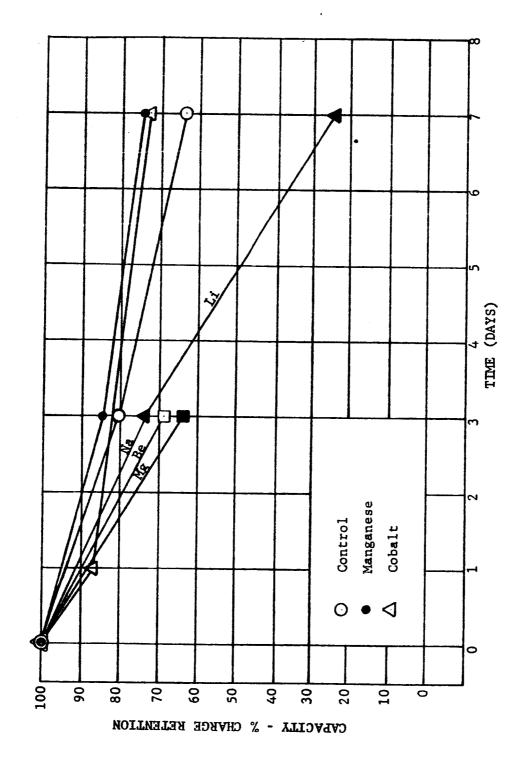






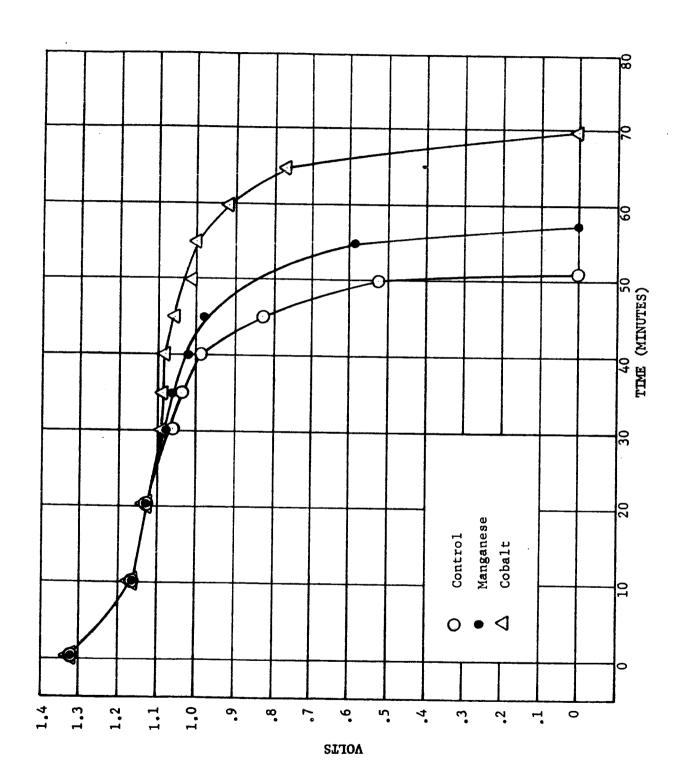


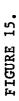
DISCHARGE CURVE (200 mA) OF CONTROL CELLS AND CELLS CONTAINING 20 ATOM % METAL ADDITIVE FIGURE 12.

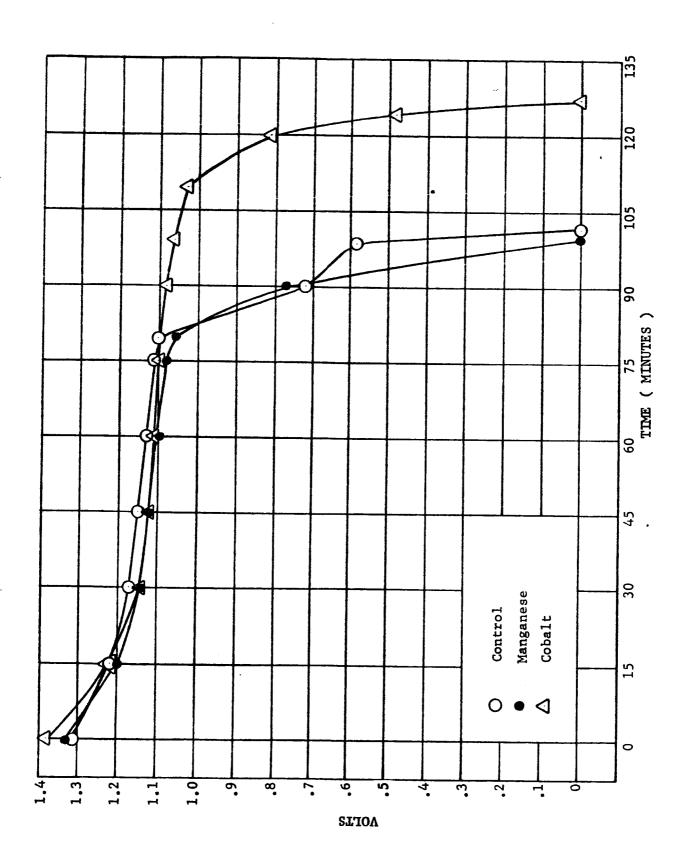


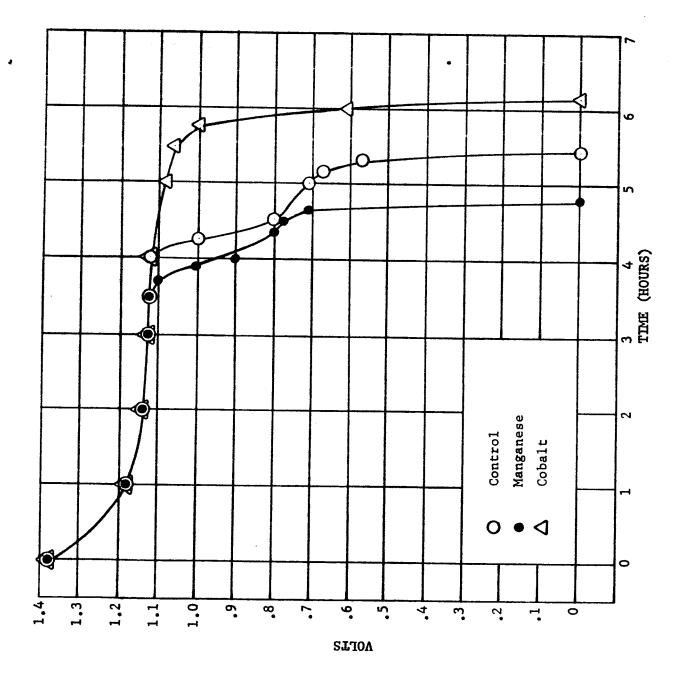
COMPARISON OF POSITIVES CONTAINING ADDITIVES WITH CONTROL POSITIVES - STABILITY AT 65°C FIGURE 13.





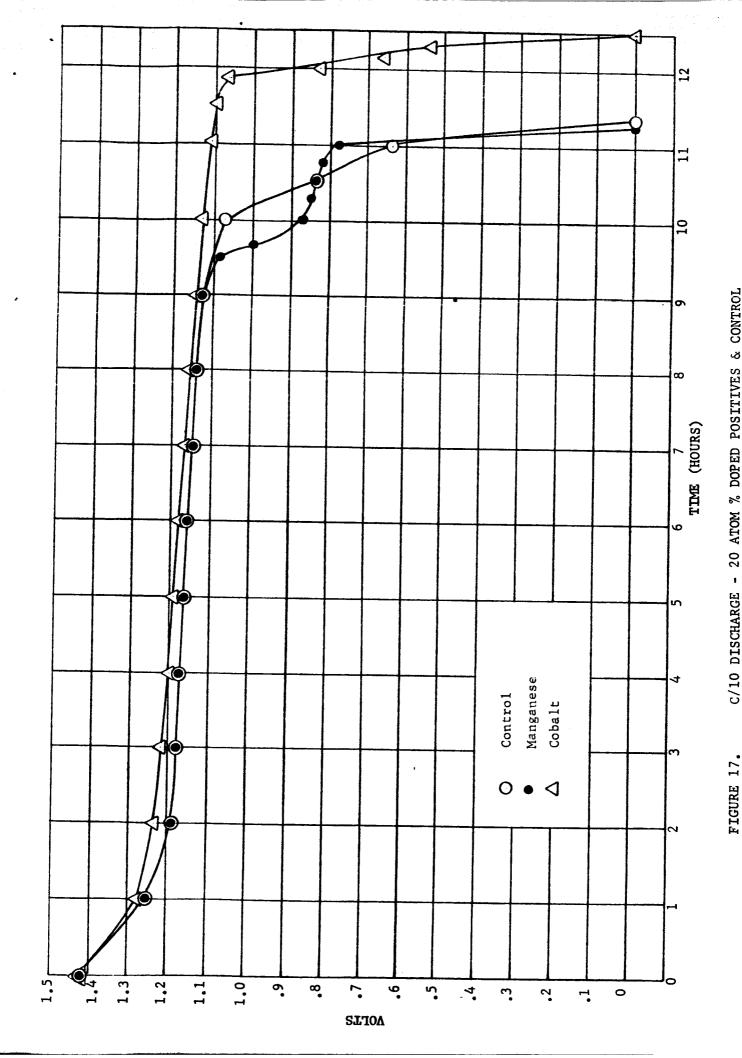




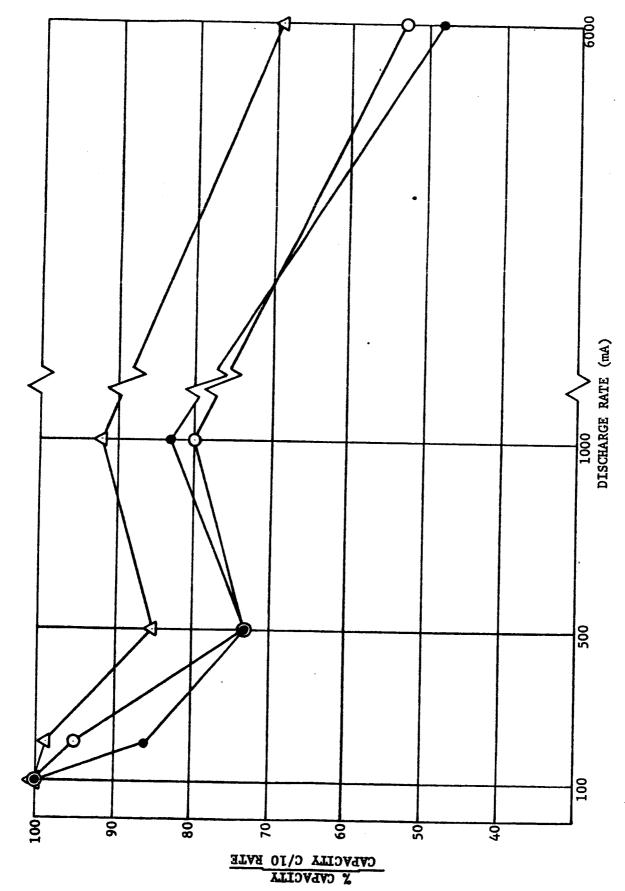


C/5 DISCHARGE - 20 ATOM % DOPED POSITIVES & CONTROL

FIGURE 16.



C/10 DISCHARGE - 20 ATOM % DOPED POSITIVES & CONTROL



UTILIZATION OF CELLS WITH POSITIVES CONTAINING 20 ATOM % METAL AND CONTROLS AT VARIOUS RAIES FIGURE 18.

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